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14. ABSTRACT The preliminary development and use of a tunable 10.6 micron CO2 laser DIAL system for the remote sensing of TATP explosive gases was experimentally studied. TATP has a large vapor pressure with strong absorption features near 3.3 micron, 8.4 micron, and 10.6 micron. Toward this end, a tunable CO2 DIAL system was constructed using a tunable 1-W CW CO2 laser, diagnostic spectrometers, 16" diameter telescope and cooled HgCdTe detectors. The backscatter lidar return from a remote retro-reflector target at a range of about 100m was used for the lidar/DIAL signal. The DIAL beam was also transmitted through a laboratory absorption cell containing an injected small sample of TATP. Detection and measurements of the TATP gas concentration in the cell were made at levels of 1 ng/micro liter. However, the concentration of TATP was found to be unstable over long periods of time due possibly to re-absorption and crystallization of the TATP vapors on the absorption cell windows. Our results also indicate that a more optimized pulsed CO2 laser DIAL system could be used for greater detection ranges, and that pulsed DIAL systems near 3.3 micron and 8.4 micron could also be used for TATP detection.					
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Final Report

Spectral Analysis for DIAL and Lidar Detection of TATP

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Executive Summary:

The preliminary development and use of a tunable 10.6 μm CO₂ laser DIAL system for the remote sensing of TATP explosive gases was experimentally studied. TATP has a large vapor pressure with strong absorption features near 3.3 μm , 8.4 μm , and 10.6 μm . As such, DIAL systems may be considered for the remote sensing of a TATP gas cloud surrounding a solid sample of TATP. Toward this end, a tunable CO₂ DIAL system was constructed using a tunable 1-W CW CO₂ laser, diagnostic spectrometers, 16" diameter telescope and cooled HgCdTe detectors. The backscatter lidar return from a remote retro-reflector target at a range of about 5 to 100m was used for the lidar/DIAL signal. The DIAL beam was also transmitted through a laboratory absorption cell containing an injected small sample of TATP. Initial DIAL results were positive and showed the detection of the TATP gas sample with moderately strong DIAL signals. Detection and measurements of the TATP gas concentration in the cell were made at sensitivity levels of 0.5 ng/ μl for a 0.3 m path. However, the concentration of TATP was found to be unstable over long periods of time due possibly to re-absorption and crystallization of the TATP vapors on the absorption cell windows. A heated cell partially mitigated these effects, but further detailed studies to control the TATP chemistry are required to better quantify our results. Our results also indicate that a more optimized pulsed CO₂ laser DIAL system could be used for greater detection ranges, and that pulsed DIAL systems near 3.3 μm and 8.4 μm could also be used for TATP detection.

A Seedling grant was awarded by AFOSR (Award FA9550-06-1-0363) via DARPA/MTO for the theoretical analysis of the lidar remote sensing of TATP explosive vapors, and modified to also include initial experimental measurements using a low cost tunable CW CO₂ laser in a DIAL system. This Final Report summarizes these studies. Several conference papers have been presented and a journal paper based upon these studies has been submitted.^[1-3]

1. Program Overview

The Differential- Absorption Lidar (DIAL) technique is a sensitive method for the long range remote sensing of molecular constituents in the ambient atmosphere.^[4-7] DIAL systems operating in the infrared have been used for monitoring the presence of major atmospheric constituents such as CO as well as trace constituents such as C₂H₂.^[7,8] In this Final Report we report on the development and use of a CO₂ DIAL system for the potential remote sensing of Triacetone Triperoxide (TATP) vapors. TATP is notable as an explosive that does not contain nitrogen, but does have a significant vapor pressure.

Our experiments were carried out using a CW CO₂ laser DIAL system in conjunction with a large optical cell which contained the target TATP gas. Lidar returns were obtained from the laser beams which passed through the cell and were reflected from a retro reflector placed at a range of 100m. The experimental results established initial experimental capability of a CW CO₂ DIAL lidar system to detect TATP and SF₆ used as a standard DIAL trace gas.^[9-17] Our results indicate that TATP can be detected by a remote CO₂ DIAL system. However the TATP concentration measured was found to be variable due to chemical instabilities and surface related absorption. Our initial results are positive but also point toward the need for better control of the TATP concentration to better quantify our DIAL remote sensing measurements.

2. Introduction to TATP.

Triacetone Triperoxide (TATP) also known as Acetone peroxide (TCAP) is an organic peroxide explosive discovered in 1895 by Richard Wolffenstein.⁴⁻⁶ The vapor pressure of TATP is 7Pa (0.052 Torr), which is about 14,000 times that of other explosives such as TNT. It is this high vapor pressure that may produce a gas plume of TATP surrounding a large sample of solid TATP, and is thus susceptible for detection using a DIAL lidar system. TATP also has a high sublimation rate and loses about 6.5% of its volume into the vapor phase in a 24 hour period.^[16]

The absorption spectrum of solid state and vapor phase TATP was measured using a FTIR spectrometer by M. Sigman. Figure 1(a) shows the measured vapor phase FTIR spectrum of TATP and Fig 1(b) shows the FTIR-ATR microscope spectrum of TATP. For the vapor phase measurements, a 5cm long cell was used at a temperature of 28 °C. The concentration of TATP in the vapor phase was estimated to be 8.28 Pa (0.062 Torr) under these conditions. For the solid phase absorption measurements, a FTIR-ATR microscope was used.

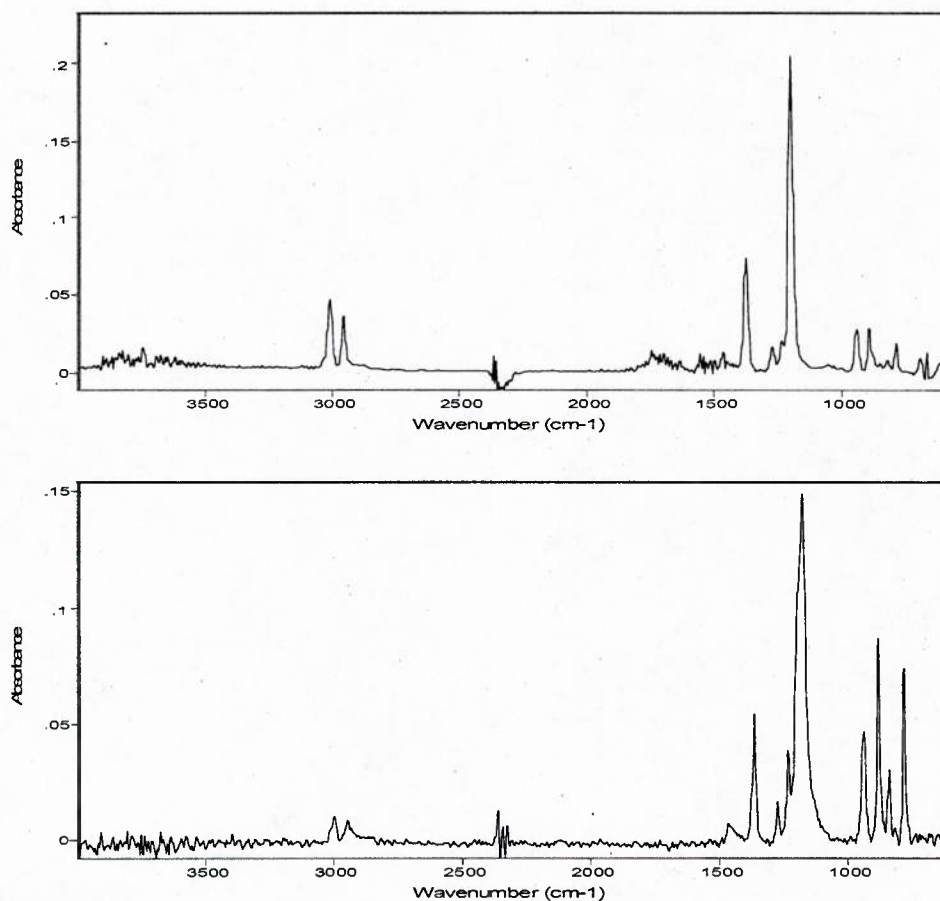


Figure 1. (a) Upper Graph: Vapor phase FTIR spectrum of TATP
(b) Lower Graph: Solid phase FTIR-ATR microscope spectrum of TATP.

As can be seen, various strong absorption lines were observed. In order to see these lines better, Fig.2 shows an enlargement of the vapor phase TATP spectrum, with the x-axis re-plotted in terms of wavelength, which is proportional to the inverse of the wavenumber values in Fig.1.

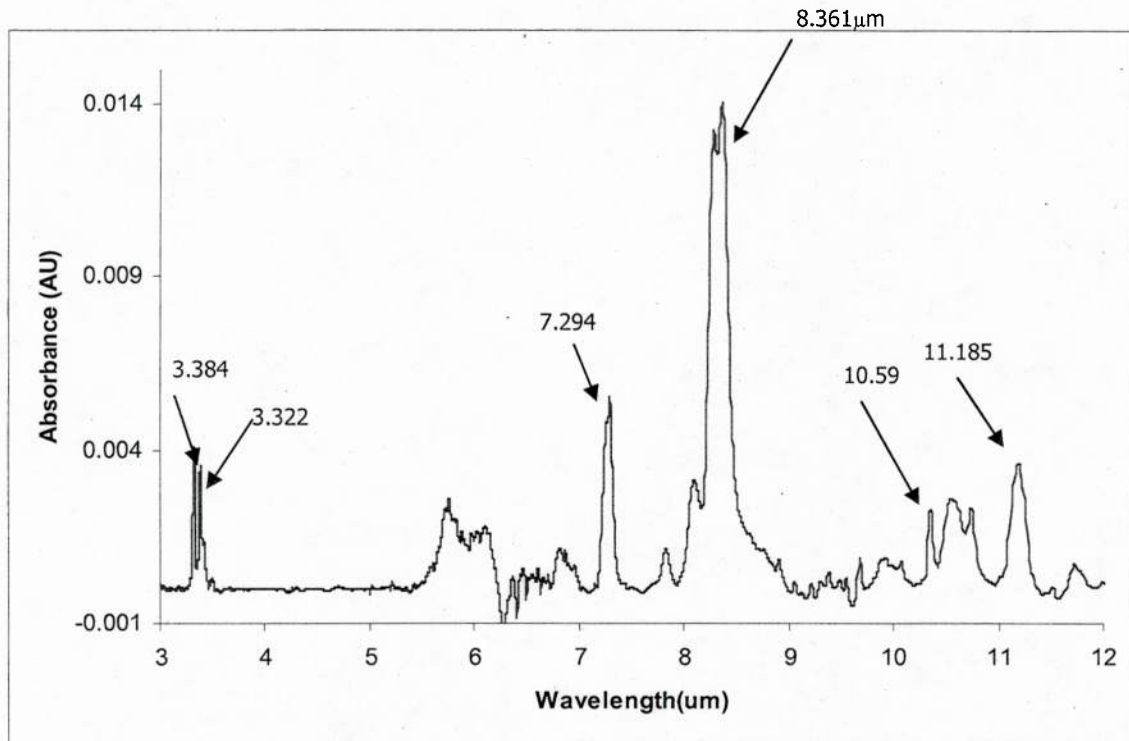


Figure 2 : Measured absorbance spectrum as a function of wavelength for vapor phase TATP (cell path length of 5cm; TATP concentration/partial pressure 8.28 Pa; 28 C)

As can be seen, the absorption lines of TATP are fairly strong with lines occurring near 8.36 μ m, 7.3 μ m, 3.38 μ m, and near 10.59 μ m. For example, the absorbance, A , of TATP in Fig. 2 at 10.59 μ m is about 0.0024. Relating the absorbance, A , to the transmission, T , as $A = -\log_{10}(T)$, and noting the Beers-Lambert relation of $T = \exp(-P_g \alpha L)$, one calculates that the attenuation coefficient, α , is 0.014 / Pa – m (or 1.75 / Torr-m), where P_g is the partial pressure of the absorbing gas in Pa, and L is the path length in meters.

It is informative to predict the absorption due to a 1-m diameter cloud of TATP. For a 1-m diameter cloud of TATP having a saturated vapor phase concentration of about 7 Pa at 25°C, the absorption for a 2-way DIAL path would be 31% for 3.32 μ m, 73% for 8.36 μ m, and 17% for the 10.59 μ m line. This is shown in Fig. 3 showing the predicted absorption of a 1-m cloud of TATP vapor. As can be observed in Fig. 3, a cloud of TATP could potentially be detected using a DIAL system at wavelength near 7.29 μ m, 8.4 μ m, 3.3 μ m, 10.6 μ m or 11.2 μ m.

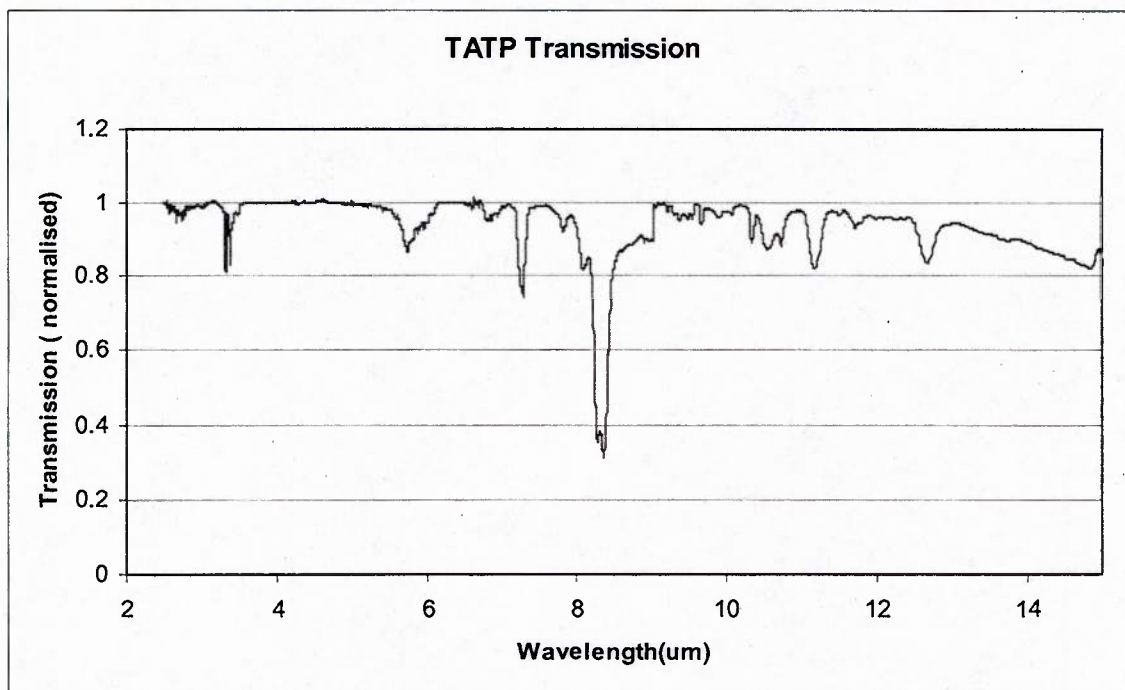


Figure 3. Predicted transmission through a 1-m cloud of TATP as a function of wavelength.

3. Experimental Setup.

We constructed a laboratory DIAL lidar system using a grating tuned CW CO₂ laser. A schematic of the DIAL lidar system is given in Fig 4.

Schematic of Laboratory DIAL LIDAR setup.

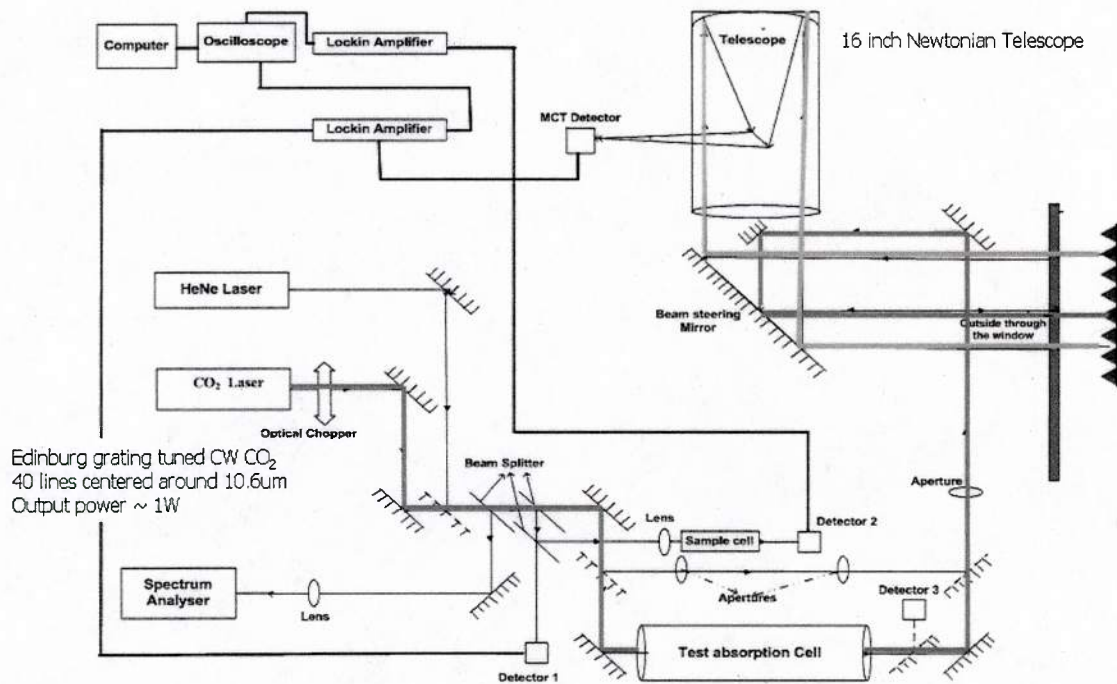


Figure 4. Schematic of laboratory Lidar DIAL setup

A grating tuned CW CO₂ laser (Edinburg Instruments, Model WL-86T) was used for producing the line tunable emission near 10.6 μ m. The laser had a CW power level of about 1W and could be tuned over about 40 different lines from 9.7 μ m to about 11.2 μ m. The output from the laser was sent through an optical chopper (SRS Model # SR540), and directed via beam-splitters toward a CO₂ laser line spectrum analyzer (Opt Eng Model # LSA 16-A), a pyro-electric detector (Eltec Model # 420-0-1491) for power monitoring, and through either an absorption cell containing SF₆ or through a Test Absorption Cell containing the TATP gas sample; the SF₆ cell was 5cm long with ZnSe windows, while the test absorption cell was 1.75m long with mylar windows and was constructed using PVC pipe. The laser beam was sampled and detected after passage through the cells, but the major portion of the beams was directed via mirrors to a large Beam Steering mirror toward targets outside our lab window or towards a retro-reflector

array target placed at a range of 5 m to 100m; the retro- reflector array target consisted of a grouping of thirty 3" diameter, gold coated retro-reflectors.

The backscattered lidar returns were collected by a 16" diameter telescope (Meade Model # DS 16) and detected by a liquid nitrogen cooled Mercury Cadmium Telluride (MCT) detector. The chopped signal was detected using lock-in amplifier (SRS, Model # SR810 DSP) and interfaced to a computer with a Labview (software) program ; a chopper frequency of 330Hz was usually used in our experiments.

A photograph of our setup and the retro-reflector array is shown in Fig 5. DIAL experiments were conducted by tuning the CO₂ laser wavelength to an "off-line" wavelength and then to an "on-line" wavelength, and deducing the concentration of the target gas from the differential absorption or different intensities of the on and offline lidar returned signals.

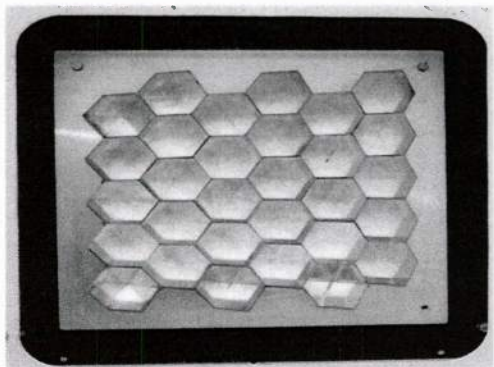


Figure 5. (a) Upper Photo: Gold coated retro reflector array; (b) lower photo: photograph of laboratory CO₂ laser DIAL system

4. DIAL calibration with SF₆.

SF_6 was used as a calibration gas for our DIAL system because of its strong absorption lines near $10.6\mu\text{m}$ and its past use by various groups.^[10,11] A 5cm long aluminum cell with ZnSe windows was used, and filled with 0.2 to 0.5Torr of SF_6 . Figure 6 shows the qualitative transmission spectra of SF_6 gas as a function of wavelength between $10\mu\text{m}$ and $11\mu\text{m}$. As can be seen the P(24) line near $10.632\mu\text{m}$ can be used as the “on-resonance” wavelength, and the R(24) line near $10.220\mu\text{m}$ can be used as the “off-resonance” wavelength for the DIAL measurements.

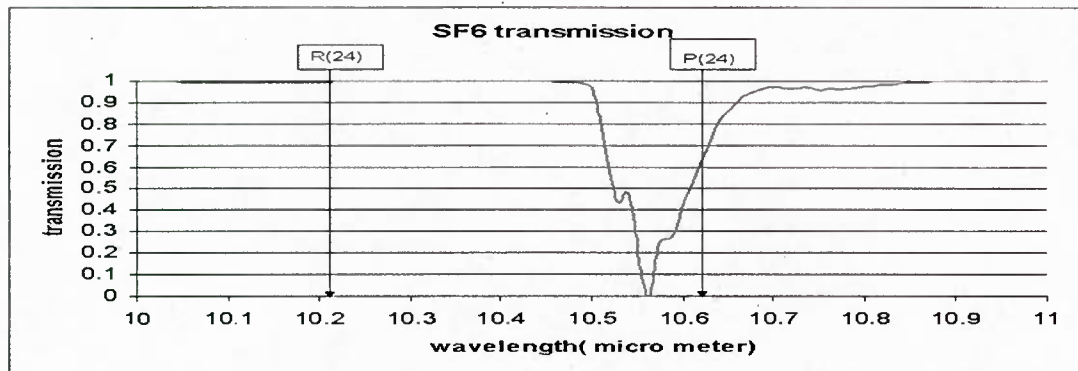


Figure 6. Qualitative transmission spectra of gaseous SF_6 as a function of wavelength near the R(24) and P(24) CO_2 laser lines..

The DIAL beam was directed through the SF_6 cell and then toward a retro-reflector array target placed at a range of 100m outside our lab window. Lidar returns as a function of time for each of the on/off resonance CO_2 wavelengths were recorded and are shown in Fig 7 for two different SF_6 concentrations. As can be seen in Fig 7, there was about 80% transmission (i.e. 20% absorption) for the case of the 0.2Torr(5cm) SF_6 case and about 50% transmission (i.e 50% absorption) for the case of the 0.5Torr (5cm) SF_6 gas with variability of 10%. These results are consistent with previous Lidar experiments using SF_6 with CO_2 wavelengths which measured an attenuation coefficient of 26/Torr-m for the 10P(24) line at $10.632\mu\text{m}$.^[9-11]

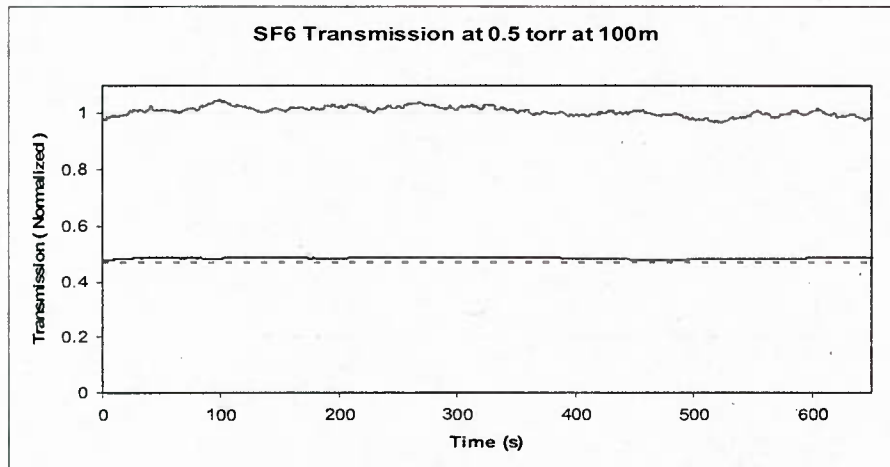
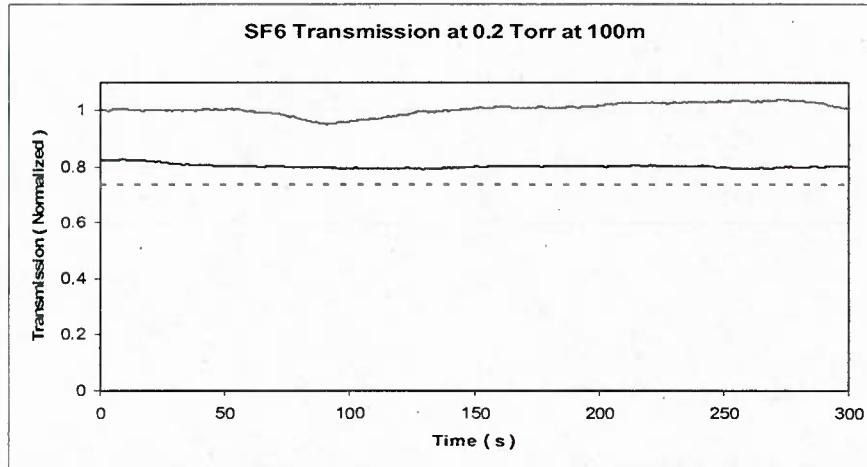


Figure 7. (a) Upper Graph: Transmission of SF₆ at 0.2Torr at 100m range as function of time. (b) Lower Graph: Transmission of SF₆ at 0.5Torr at 100m range as function of time. Dotted line is predicted transmission.

5. DIAL Lidar detection of TATP gas.

The CO₂ DIAL system was used with TATP gas in the laboratory cell. In this case, the test absorption cell in Fig. 4 was a PVC 175cm long plastic pipe cell with mylar windows to transmit the CO₂ wavelengths. The cell had injection ports on the side for the delivery of TATP into the cell. The expected TATP transmission spectrum for a

1.75m path of TATP vapor at a concentration or partial pressure of 4.3 Pa was calculated from the FTIR spectra of TATP of Fig. 1 and is shown in Fig 8. As can be seen from Fig. 8 the absorption between the offline and the online resonance DIAL wavelength should be about 10%.

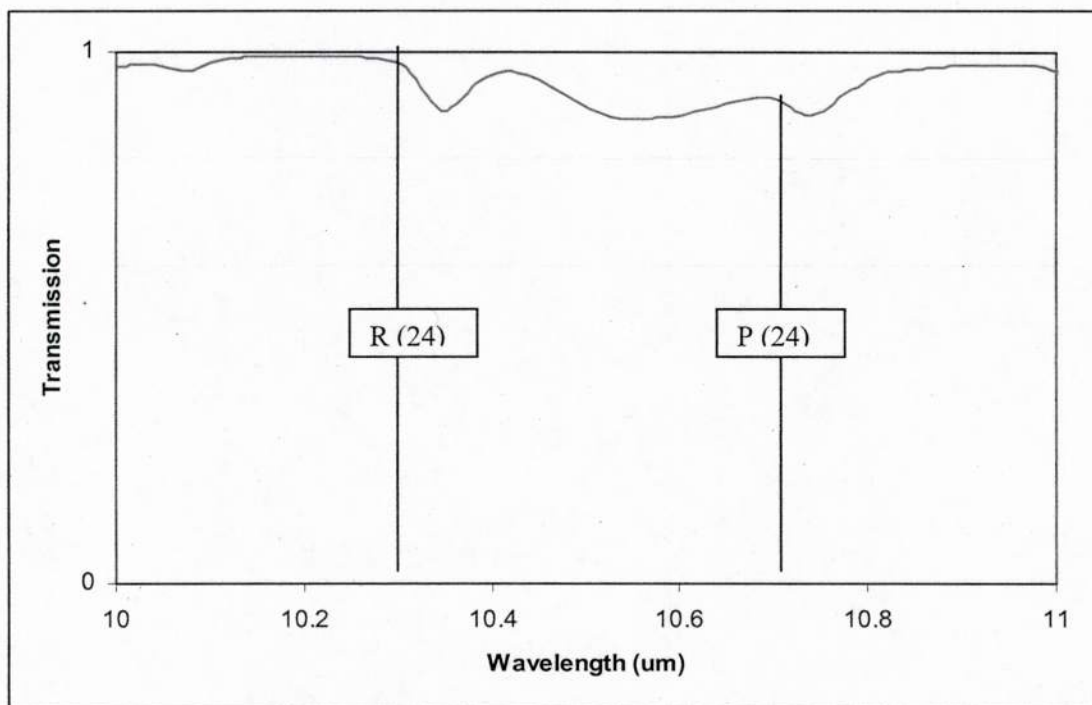


Figure 8. Expected transmission spectra of TATP for a 175cm path length.

Initial DIAL experiments were conducted by passing the DIAL laser beam through the 1.75m Test Absorption Cell, toward the distant retro-reflector array target, and backscatter detected by the telescope and MCT detector. In this case, the target was at a range of 5 m to increase the S/N due to losses experienced directing the beam through the cell using the beam splitters. A small (about 1 mg) sample of TATP was prepared and left in a chloroform (CHCl_3) solvent. The sample was then injected via a syringe into the absorption cell and allowed to mix for several minutes. During this time, the CO_2 laser was operating on the “on-resonance” P(24) line so that absorption due to TATP could be observed. However, in this case negligible absorption was observed. It was determined that the TATP was difficult to disperse evenly throughout the entire cell, and that possibly stratification could have occurred even though large (>200cc) syringe pumps were used to circulate and mix the gas inside the cell. Calculations indicate that 1 mg of TATP within the cell (volume of $17,500 \text{ cm}^3$) would produce a concentration of about $0.05 \text{ ng}/\mu\text{l}$, and produce only about 2% absorption of the single-pass online beam. Our measurements are consistent with these limitations. It was determined that use of a larger sample size was not prudent. It should be added that the conversion between partial pressure P_g and concentration is $1 \text{ Pa} = 0.088 \text{ ng}/\mu\text{l}$ at 25°C and $1 \text{ Pa} = 0.077 \text{ ng}/\mu\text{l}$ at 70°C using the ideal gas law.

To increase the TATP vapor pressure and the optical path length inside the absorption cell a second (different) glass absorption cell was used that could be heated so that the vapor pressure and the concentration of TATP increases; the cell was 30cm long and used Mylar windows. The cell was heated to a temperature near 70°C by using heating tape wrapped around the 20cm central portion of the cell. A 200 μl TATP sample (concentration of 1 μg in 1 μl of CHCl_3 solution) was injected into the cell, and the “on-resonance” DIAL laser beam transmission through the cell was recorded. Figure 9 shows our measured transmission signal as a function of time over the period when the TATP was injected. As can be seen, there was about 10 % absorption when the gas was injected.

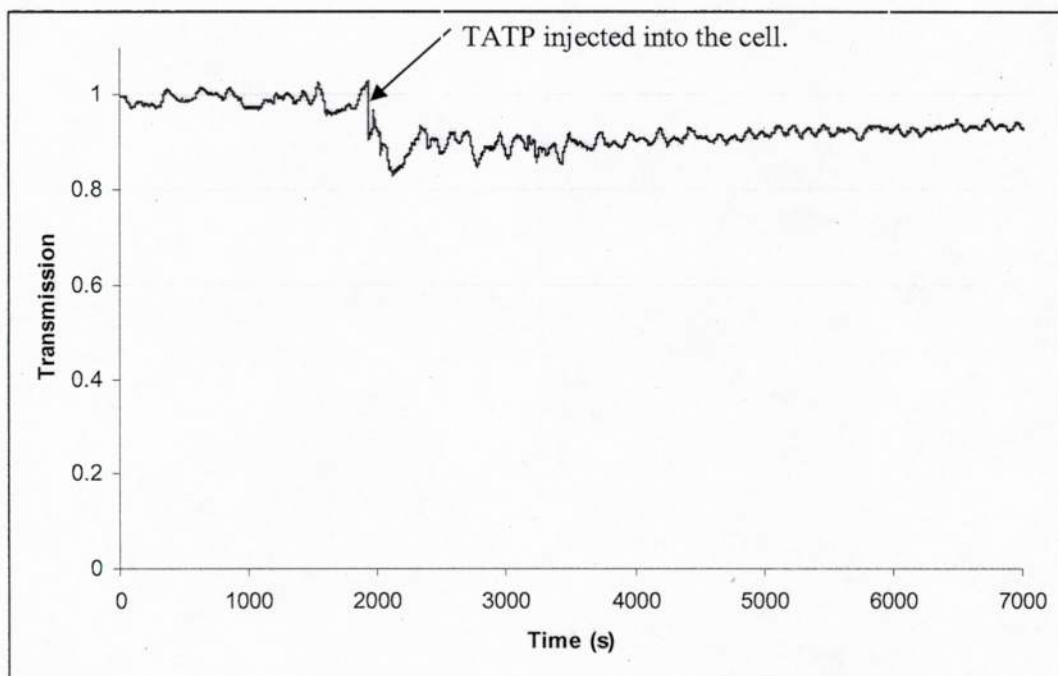


Figure 9. Online DIAL transmission with injection of TATP into a heated 30cm long absorption cell.

Figure 10 shows the measured concentration of TATP by our DIAL lidar setup inside the heated absorption cell as the TATP was injected. The measurements indicated TATP concentration of approximately 1.8 ng/ μl . In order to better quantify the concentration inside the cell, a small sample of the gas inside the cell was obtained using a precession volumetric syringe and the sample injected into a calibrated Mass-spectrometer system (ThermoFinnigan Trace DSQ). Two different IMS readings were obtained over a period of about 20 minutes and yielded values of 0.72 and 0.99 ng/ μl .

Our DIAL measured value is consistent with that measured by the Mass-spectrometer with some variability during the time of introduction of the sample into the cell.

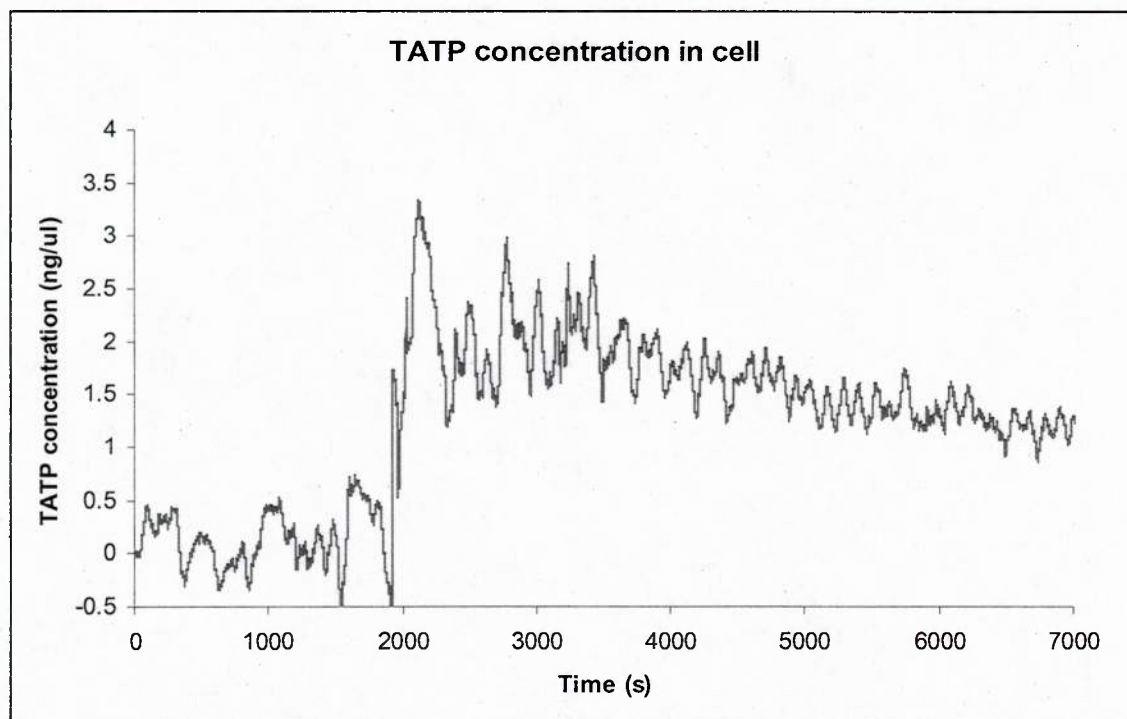


Figure 10. DIAL measured TATP concentration inside a heated 30cm long absorption cell as function of time.

It was noticed that there were often long term (few minutes) temporal changes in the DIAL signal. The observed variation in transmission may be due to chemical induced changes in TATP with temperature and time as reported by other groups.⁶⁻¹⁰ Upon further examination of the cell, it was found that TATP crystals were being formed upon the unheated Mylar windows. Attempts to heat the windows slightly were not successful. We are now working on a new absorption cell with ZnSe windows that can be heated uniformly including the windows. We anticipate that such a cell will be able to better stabilize the concentration of TATP within the absorption cell. Finally, field tests are being planned to test our CO₂ DIAL system for detection of a potential TATP plume surrounding a large sample of TATP.

6. Conclusion

A tunable CO₂ DIAL system has been developed for the first time to our knowledge for the potential detection of TATP gas clouds. The system has been used to

measure gas samples of SF₆, and has shown initial absorption measurements of samples of TATP contained within an enclosed optical absorption cell. DIAL/Lidar returns from a remote retro-reflector target array were used for the DIAL measurements after passage through the laboratory cell containing the TATP gas. DIAL measured concentrations agreed well with those obtained using a calibrated Ion Mobility Spectrometer. DIAL detection sensitivity of the TATP gas concentration in the cell was about 0.5 ng/μl. However, the concentration of TATP was found to be unstable over long periods of time due possibly to re-absorption and crystallization of the TATP vapors on the absorption cell windows. A heated cell partially mitigated these effects, but further detailed studies to control the TATP chemistry are required to better quantify our results. We plan to extend these preliminary one-way DIAL measurements to that of a two-way DIAL measurement by placing the absorption cell outside the laboratory window, if the TATP concentration within an external cell can be controlled. In addition, a more optimized high power pulsed CO₂ laser DIAL system could be used for greater detection ranges, and that pulsed DIAL systems near 3.3 μm, 7.3 μm and 8.4 μm could also be used for TATP detection.

7. Personnel Supported: A graduate student, Avishekh Pal was supported by this grant. He plans to graduate with a Ph.D. in the Fall of 2008. In addition, a small amount of support was for P.I. Dennis Killinger.

8. Publications: A paper has been submitted.¹

9. Patents: A patent was applied for the use of a CO₂ DIAL system for the detection of TATP. Application 11/560,192, dated 11/15/2006: Laser Remote Detection of TATP Explosives (USF Ref: 05B121PRC/Killinger et. al.)

10. Acknowledgement: We want to acknowledge the collaboration of Prof. Michael Sigman and C. Douglas Clark from the University of Central Florida for FT-IR spectral measurements of TATP and for preparing the samples of TATP along with Mass-Spec calibration of these samples during the DIAL measurements.

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Final Progress Survey Profile Report

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Page One

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The preliminary development and use of a tunable 10.6 micron CO2 laser DIAL system for the remote sensing of TATP explosive gases was experimentally studied. TATP has a large vapor pressure with strong absorption features near 3.3 micron, 8.4 micron, and 10.6 micron. Toward this end, a tunable CO2 DIAL system was constructed using a tunable 1-W CW CO2 laser, diagnostic spectrometers, 16" diameter telescope and cooled HgCdTe detectors. The backscatter lidar return from a remote retro-reflector target at a range of about 100m was used for the lidar/DIAL signal. The DIAL beam was also transmitted through a laboratory absorption cell containing an injected small sample of TATP. Detection and measurements of the TATP gas concentration in the cell were made at levels of 1 ng/micro liter. However, the concentration of TATP was found to be unstable over long periods of time due possibly to re-absorption and crystallization of the TATP vapors on the absorption cell windows. Our results also indicate that a more optimized pulsed CO2 laser DIAL system could be used for greater detection ranges, and that pulsed DIAL systems near 3.3 micron and 8.4 micron could also be used for TATP detection.

8. Archival Publications (published) during reporting period:

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